SPECIFICATION

MAGNETIC THIN FILM AND PRODUCTION METHOD THEREFOR

Technological Field

The present invention relates to a magnetic thin film and a manufacturing method therefor; in particular, the present invention relates to a manufacturing method for thin magnetic films which is capable of stably producing an iron nitride film having high saturation. The present invention may be advantageously applied to the magnetic heads or the like of magnetic discs having high recording density.

Background Art

Iron nitride thin films, in particular, $Fe_{16}N_2$ thin films, have a particularly large saturation among magnetic materials, and have attracted attention as new materials which may be finely worked for magnetic head materials and the like; however, because these materials are not stable with respect to heat, the formation of thin films under high temperatures with such materials is impossible, and the stable formation of thin films having superior characteristics has become difficult. However, in recent years, Omuro et al. (Journal of the Japanese Society of Applied Magnetism, 14, 701, 1990) have made it possible to produce a monocrystalline iron nitride film ($Fe_{16}N_2$) using the MBE method, and since the enormous value of the magnetic moment

of such a thin film has been confirmed, these materials have again attracted attention, and a manufacturing method which is capable of application has been anticipated.

However, the following problems exist in this manufacturing method: (1) a special substrate ($In_{0.2}Ga_{0.8}As$) is required, (2) the film formation rate is slow (0.05Å/sec or less), (3) the critical film thickness in the single phase state of the stably formed $Fe_{16}N_2$ is small (1000Å or less), and (4) the process of the nitridization of the Fe from the gas phase is unclear, and instabilities remain in the thin film formation; thus, the current state of affairs is such that there are great obstacles to the application of iron nitride thin films.

In light of the above circumstances, the present invention has as an object thereof to provide a magnetic thin film formation method which is capable of rapidly and stably forming an iron nitride thin film present in a single phase state while at a film thickness of 1000Å or more, and which does not not require a special substrate. Furthermore, the present invention has as an object thereof to provide a magnetic thin film having high saturation and a low coercive force.

Disclosure of the Invention

With respect to a stable formation method for iron nitride thin films, the attention of the present inventors was drawn to the use of a reactive plasma of N_2 gas; they have developed

experiments relating to standardized plasma analysis and the process of nitridization by means of a vapor deposition method and sputtering method, and have investigated the relationship between the plasma and the iron nitride thin film phase which is synthesized. The present inventors have selected the plasma conditions and produced the iron nitride thin film on a substrate by means of an opposed-target DC sputtering method have made clear the growth conditions of α'' - $Fe_{16}N_2$, and they have considered the relationships between the phase, the structure, and the saturation. The present invention was completed based on these insights.

That is say, the magnetic thin film of the present invention is characterized in comprising an iron nitride thin film which is formed on a substrate by means of an opposed-target DC sputtering method employing reactive sputtering with N_2 gas.

Furthermore, the present invention is characterized in that by means of the opposed-target DC sputtering method, an iron (α - Fe) thin film and a iron nitride thin film are alternately layered on a substrate.

Furthermore, the magnetic thin film manufacturing method in accordance with the present invention is a manufacturing method for iron nitride thin films which employs an opposed-target DC sputtering method, characterized in that iron nitride thin film is formed on a substrate by introducing Ar and N_2 gases into a

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film formation chamber, and applying DC power to an iron target within the Ar and N_2 gas atmosphere.

In a preferred mode of the manufacturing method of the present invention, the flow rate of the N_2 is within a range of 8 - 25% of the total gas flow rate.

Furthermore, it is preferable that the electron temperature during formation of the iron nitride thin film be within a range of 0.01 - 1 eV, and that the electron density be within a range of $1 \times 10^9 - 1 \times 10^{10}$ cm^{-3}.

Furthermore, in a preferred mode of the present invention, the substrate has an iron thin film formed thereon as a base layer.

Additionally, in the present invention, it is preferable that after the formation of the iron nitride thin film, heat treatment be conducted in a vacuum, and it is preferable that the heat treatment be such that the temperature is within a range of 100 - 180° C, and the treatment is conducted for a period within a range of 1 - 3 hours.

Function

By means of the present invention, it is possible to rapidly and stably form an iron nitride thin film having an extremely large saturation Ms, by means of employing an opposed-target DC sputtering method. Additionally, by means of setting the electron temperature and electron density during film formation to within ranges of, respectively, 0.01 - 1 eV and 1 x

10^9 - 1 x 10^{10} cm^{-3}, it is possible to further increase the uniformity and stability of the characteristics of the film, such as saturation and the like.

By means of setting the flow rate of the N_2 gas to within a range of 8 - 25% of the total gas flow rate, it is possible to more stably form the single phase α crystalline phase.

Furthermore, it is preferable that an iron thin film (α - Fe) be formed as a base layer on the substrate. By means of employing such a substrate, the monocrystalline nature of the thin film is further increased.

Additionally, it is preferable that after the iron nitride thin film formation in the present invention, heat treatment be carried out in a vacuum, and it is preferable that the conditions of the heat treatment be such that the temperature is within a range of 100 - 180° C, and the treatment is carried out for a period of time within a range of 1 - 3 hours. By means of conducting heat treatment, it is possible to produce an α" crystalline phase ($Fe_{16}N_2$) and to further increase the saturation.

By providing a layered structure of α - Fe and iron nitride in the magnetic thin film in accordance with the present invention, it is possible to reduce the coercive force.

Brief Description of the Drawings

Figure 1 is a graph showing the relationship between the X-ray diffraction pattern of the iron nitride thin film after film

formation, and the flow rate ratio of the N_2 gas during film formation.

Figure 2 is a graph showing the X-ray diffraction pattern of the iron nitride thin film after heat treatment.

Figure 3 is a graph showing the changes in the X-ray diffraction pattern of the iron nitride thin film immediately after film formation and after heat treatment.

Figure 4 is a graph showing the relationship between the amount of N contained in the α" and α' phases, and the flow rate ratio of the N_2 gas during film formation.

Best Mode for Carrying Out the Invention

Hereinbelow, the present invention will be explained in detail based on embodiments; however, it is of course the case that the present invention is in no way limited to the embodiments described.

(Embodiment 1)

The film formation of the iron nitride thin film was conducted by means of an opposed-target DC sputtering method under conditions such that the total gas pressure was 5 mTorr, the total gas flow rate was 5 ccm, and the N_2 gas flow rate ratio was within a range of 2 - 25%. A MgO (001) monocrystalline substrate was employed as the substrate, and prior to film formation, the substrate was subjected to heat treatment for a period of 2 hours at a temperature of 200° C

within a chamber, and was then allowed to cool to room temperature.

First, an iron base layer having a thickness of 50Å was formed in a Ar atmosphere (at a film formation rate of 33Å/min), and on this, an iron nitride thin film having a thickness of 3000Å was formed in a Ar and N_2 gas atmosphere, at a film formation rate of 200Å/min. Film formation was conducted under conditions such that the electron temperature Te was equal to 0.3 eV and the electron density Ne was equal to 1×10^{10} cm^{-3}; these were determined on the basis of plasma diagnostic results.

After film formation, heat treatment was conducted by temporary exposure of the thin film to the atmosphere, and then heating at a temperature of 150° for a period of 2 hours at a pressure of 5×10^{-6} Torr in a vacuum furnace.

The structural analysis was conducted by means of X-ray diffraction (CoKα rays). Furthermore, the amount of N contained in the thin film as determined from the relationships between the lattice constant and the N concentration at the nitrogen marten-site which were disclosed by Jack (Proc. Roy. Soc., A208, 200, 1951).

In Figure 1, the X-ray profiles of a series of iron nitride thin films produced with N_2 flow rate ratios within a range of 2 - 25%, immediately after film formation, are shown. Furthermore, in the Figure, the N concentration corresponding to each diffraction line is also shown.

In the profiles on the large angle side in which 65° < 2θ < 80°, diffraction rays were only observed from the (002) surface of the nitrogen marten-site α' phase (hereinbelow abbreviated to α(002)), in which N atoms were arranged irregularly in the body-centered cubic lattice of the iron nitride.

It was determined that the thin film produced at N_2 gas flow ratios of 5% or less comprised two phases, α - Fe and the α' phase, while thin films produced at 8% or more comprised only the single α' phase. Additionally, in concert with the increase in the N_2 gas flow rate ratio, the diffraction rays from the α' (002) moved in the direction of the smaller angles, from 75° to 68°, and this corresponds to an increase in the amount of N contained in the α' phase.

In Figure 2, X-ray profiles of the thin films shown in Figure 1, obtained after heat treatment, are shown. It can be seen from the profiles on the large angle side that the diffraction rays from the α' which was grown in a nearly single phase state immediately after film formation are clearly separated from the diffraction rays from the α' phase (002) surface containing approximately 2 - at% of N, and the diffraction rays from the α" phase (004) surface (hereinbelow shortened to α" (004)) and the α' phase (002) surface containing approximately 11 at% N. Additionally, furthermore, in the small angle side profiles, the diffraction rays from the α" (002) surface (hereinbelow shortened to α" (002)), which are the

diffraction rays unique to the α" phase, are clearly observed in the thin films produced with N_2 gas flow rate ratios of 12% or greater during film formation. These diffraction rays are produced when a regular arrangement of N atoms is selectively caused in the $Fe_{16}N_2$ phase. From this, it is thought that, although a body-centered cubic structure is maintained in the thin films prior to heat treatment, the N atoms are not in a regular arrangement, and that such a regular arrangement of the N atoms is promoted by heat treatment, thus producing the α" phase.

It was confirmed that the α" layer is essentially not formed at heat treatment temperatures of 100°C or less.

In Figure 3, in order to consider in greater detail the state of the phase separation from the α' to the α" + α' seen in Figure 2, the patterns of change in the diffraction rays at large angles, before and after heat treatment, in thin films produced under various flow rate ratios are shown. In those prior to heat treatment, it can be seen that the α' single phase is present in all thin films (the single dotted line in the Figure). The changes after heat treatment are such that, using as an example the profile of the thin film produced using a N_2 gas flow rate ratio of 12%, the α' phase prior to heat treatment, which is assumed to contain 8.5 at% N, is divided after heat treatment into the α" and α' phases having a large amount of N contained, at 10.6 at%, and the α' phase having a small amount of N contained, at 1.3 at%. Similar trends in the

changes were observed in the thin films produced using all other N_2 gas flow rate ratios. In the thin film produced using a N_2 gas flow rate ratio of 25%, diffraction rays from a γ' phase were observed. On the other hand, in the thin films after heat treatment, the intensity ratio $I_{\alpha'(002)}/I_{\alpha''(004) + \alpha'(002)}$ between the diffraction rays from the α' phase (002) surface containing approximately 2 at% N, and the diffraction rays from the α'' (004) and a'(002) having the $Fe_{16}N_2$ stoichiometric composition (approximately 11 at%) decreases as the N_2 gas flow rate ratio increases; this is believed to show that the amount of α'' phase containing 11 at% of N which is generated in the film is increasing.

In Figure 4, based on the X-ray profiles shown in Figures 1 and 2, the amounts of N contained in each phase are shown with respect to the N_2 gas flow rate ratio during film formation. Immediately after film formation, the amount of N contained increases monotonically from 2.5 to 12.5 at% in concert with an increase in the N_2 gas flow rate ratio. On the other hand, as a result of the heat treatment, in the films produced using N_2 gas flow rate ratios of from 10 to 16%, the α' phase (7.5% - 11 at% N) prior to heat treatment is divided into an α'' containing a large amount of N and an α' containing a small amount of N. The N concentration within the α'' phase remains in the stoichiometric ratio of the $Fe_{16}N_2$ compound as the N_2 gas flow rate ratio increases, and shows a tendancy to be essentially saturated. Furthermore, in the thin films formed using a N_2 gas

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flow ratio of 25%, as a result of heat treatment, a γ' - Fe_4N phase (stoichiometric composition 20 at%) is formed.

As described above, in accordance with the present invention, it was confirmed that it is possible to form an iron nitride thin film having a high saturation. In particular, when a N_2 flow rate ratio of 8% or more is employed, it is possible to form a single phase α' phase, and it is thus possible to form an iron nitride thin film having more stable characteristics. Additionally, it was learned that by means of conducting heating treatment, an α'' phase having a higher saturation was formed.

(Embodiment 2)

In the same manner as in Embodiment 1, iron thin films having a thickness of 30Å and iron nitride thin films having thickness of 100Å were alternately layered on a MgO substrate so that 5 of each type of layer was present. The N_2 gas flow rate ratio during deposition of the iron nitride thin film was 20%.

After this, heat treatment was conducted in a vacuum furnace at a pressure of 5×10^{-6} Torr or less, at a temperature of 150°C, and for a period of 2 hours.

When the coercive force of the magnetic thin films obtained was measured, it was determined to be 1.0 Gauss, and it was thus learned that by means of employing the present embodiment, it is possible to obtain magnetic thin films having extremely low coercive forces.

Industrial Applicability

As described above, in accordance with the invention as stated in claim 1, it is possible to provide magnetic thin films having high saturation. Additionally, by means of the invention as stated in claim 2, it possible to provide magnetic thin films having low coercive force.

By means of the magnetic thin film manufacturing method as stated in claim 3, a high speed film formation of 200Å per minute is possible, and moreover, it is possible to produce iron nitride thin films which are single phase and have high saturations even when extremely thick in comparison with those conventionally obtainable, at 3000Å.

Additionally, in accordance with the invention as stated in claim 8, it is possible to produce a α" phase having higher saturation.

By means of the present invention, it is possible to provide thin film magnetic heads appropriate for ultra high recording densities.